ORNL 1155
Instrumentation

MONITORING OF LIQUIDS FOR RADIOACTIVITY

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Health Physics Division

MONITORING OF LIQUIDS FOR RADIOACTIVITY

W. M. Hurst

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OAK RIDGE NATIONAL LABORATORY
Operated by
CARBIDE AND CARBON CHEMICALS DIVISION
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ABSTRACT

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The development of an instrument to monitor continuously drinking water for alpha-beta-gamma radioactivity in solution has progressed so that a preliminary design and some test results can be presented. Two devices are described: (1) the continuous flow water cell with the radiation detector assembly, and (2) an instrument, for calibrating the water cell and detector response, which instrument can also be used to measure the activity of liquid samples. A Geiger counter is surrounded but not in contact with a rapidly rotating cylindrical wall of water of one cm radial thickness. K^{40} naturally present in body fluids of the human being is detected easily. Sr^{89} activity in water is detected at a concentration of 1×10^{-6} $\mu\text{c/cc}$, Tl^{204} at a concentration of 3×10^{-6} $\mu\text{c/cc}$. Alpha radiation (from Pu^{239}) is detected at 4×10^{-5} $\mu\text{c/cc}$. Operation for one hour with a solution having a Sr^{90} (plus Y^{90} in equilibrium) activity of 10^{-1} $\mu\text{c/cc}$ resulted in no measurable contamination of the counter tube.

INTRODUCTION

The liquid monitoring radiation detection apparatus to be described results from a need to monitor continuously the radioactivity in drinking water at the lowest assigned maximum permissible level. The information provided by such monitoring would be of value to persons concerned with public health, radiological protection, and radiochemical process controls. During the development emphasis was placed upon drinking water problems; however, other applications of the apparatus include: measurement of naturally occurring unstable isotopes in excreted or contained body fluids of the human being, radioactive gas concentrations, counting of activity of a solid upon the inner surface of a cylinder, and the rate of plating out of activity from a solution.

The use of water for drinking, domestic, and industrial purposes normally requires some degree of water purification and quality control.^(1, 2) For instance, drinking water for a community is usually processed so that harmful or undesirable bacteria, chemicals, and particulate matter are largely eliminated. Further processing to provide pure water would in most cases involve an unjustifiable expense because impurities present in sufficiently small amounts are considered to be relatively harmless to the human being. The U. S. Public Health Service Drinking Water Standards include recommended values for the maximum permissible concentration of some undesirable chemical substances in drinking water. These values range from 1×10^3 $\mu\text{g/cc}$ to 1×10^{-3} $\mu\text{g/cc}$, as seen in Table 1.

Since unstable isotopes emit nuclear radiations capable of

TABLE 1
MAXIMUM PERMISSIBLE CONCENTRATION IN DRINKING WATER
(incomplete listing)

SUBSTANCE	ug/cc	uc/cc
Total solids (where only one drinking water supply is available)	1×10^3	-----
Total solids (where there is a choice of supplies)	5×10^2	-----
Magnesium	1.25×10^2	-----
Zinc	1.5×10^1	-----
Copper	3×10^0	-----
Lead	1×10^{-1}	-----
Arsenic	5×10^{-2}	-----
Phenolic compounds (as phenol)	1×10^{-3}	-----
Carbon ¹⁴	6.7×10^{-4}	3×10^{-3}
Molybdenum ⁹⁹	3.0×10^{-5}	1.4×10^{-1}
Radium ²²⁶	4×10^{-8}	4×10^{-8}
Strontium ⁸⁹	2.6×10^{-9}	7×10^{-5}
Strontium ⁹⁰ (in equilibrium with Y ⁹⁰)	5×10^{-9}	8×10^{-7}
Phosphorus ³²	7×10^{-10}	2×10^{-4}

U. S. Public Health Service
Drinking Water Standards.

National Committee on
Radiation Protection,
Subcommittee on Internal Dose.

damaging tissue by ionization, they constitute a health hazard irrespective of their toxic properties. The lower limit of radiation exposure of the human being is determined by cosmic radiation and unstable isotopes in nature. For example, there are about 150 grams of potassium in the body of an adult. Potassium in nature consists of a mixture of the isotopes K^{39} , K^{40} , and K^{41} , the unstable isotope K^{40} constituting about 0.012% of the aggregate. In terms of activity, there is approximately 0.12 microcurie of K^{40} in an adult. The radiation from K^{40} is a mixture of beta particles having energies up to a maximum of 1.36 mev and gamma rays of 1.462 mev, and is one of the larger factors in assessing the normal radiation exposure of the human being.

The National Committee on Radiation Protection of the National Bureau of Standards has established a subcommittee (Committee on Internal Dose), specifically charged with the responsibility of recommending maximum permissible concentrations. Provisional levels for radioactivity in drinking water range from 2×10^{-1} microcurie/cc for tritium (H^3) to 8×10^{-7} microcurie/cc for strontium and yttrium ($Sr^{90} + Y^{90}$) for beta and/or gamma emitters. These levels depend upon such factors as the nature and energy of the radiation, isotopic half-life, retention within the body, and selective absorption by various parts of the body and their respective radiosensitivities. The maximum permissible concentration is not a simple function of any one of the various factors. As a result, drinking water should be monitored for the lowest maximum permissible concentration for that particular isotope. Included in Table 1 are some of the maximum permissible concentrations established by the Committee on Internal Dose (for a complete listing to date, the

Committee on Internal Dose should be contacted). These values are also shown in $\mu\text{g/cc}$ to provide a comparison of magnitude with the U. S. P. H. S. Drinking Water Standards.

The contamination of water with radioactive isotopes is not too common; however, there are some water sources⁽³⁾ where the activity level is readily measurable due to the leaching out of the unstable isotopes near the earth's surface. A more practical problem relates to the operation of large nuclear reactors and the radiochemical processing of unstable isotopes, where careful measurements are made of effluents to safeguard against the discharge of excessive amounts of radioactive materials into the public domain. For instance, where a radioactive waste discharge is to be considerably diluted by a large river, the effluent measurement is made at a relatively high level. Subsequent checking of the water downstream for radioactivity usually requires concentration of the sample to obtain a significant reading. Where the drinking water ingestion time may be limited (due to emergency conditions) to a few months, weeks, or days, the maximum permissible level of activity may be considerably higher. With this in mind, it is feasible to consider operation of a continuous flow drinking water monitor to alarm at the level assigned for short-term ingestion and to concentrate a sample of drinking water at appropriate intervals for the long-term ingestion low level measurements.

PRIOR ART

A report (K-837) in preparation at the K-25 Plant, Oak Ridge,

Tennessee, describes a continuous flow water monitor. The system includes three mica end window GM counters positioned immediately above a tray containing a $3/4$ in. depth of running water. The three counters have a combined background counting rate of about 60 c/m (shielded) and a total beta window area of about 3 in.². A counting rate circuit records on a continuously moving chart.

A report (HW-9584) by P. L. Eisenacher, Radiological Sciences Department, Hanford Works, Richland, Washington, describes a continuous flow water monitor of liquids having activity somewhat greater than is represented by the drinking water tolerances. The radiation detector is a GM beta counter tube, positioned vertically in the center of a falling cylindrical curtain of water formed by many water streams each about $3/16$ in. diameter. The counting rate is recorded on a strip chart recorder.

Satisfactory radiological water monitoring control instruments consisting of jacketed thin wall GM counter tubes with necessary components are now available commercially*.

DESCRIPTION AND DISCUSSION OF APPARATUS

Continuous Flow Water Monitor

Figure 1 shows a simplified section view of the water cell, radiation detector, and lead shielding assembly. The water cell (A) surrounds the radiation counter (B) with an air spaced wall of water (C). The only beta absorber between the water wall and the sensitive region of the counter is a $3/16$ in. thick annular air gap, and the counter wall.

*Information regarding sources of supply of these instruments can be obtained by writing to the Radiation Detection Instrument Branch, Division of Biology and Medicine, 1901 Constitution Avenue, Washington 25, D. C.

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Fig.1.- Simplified Section View Of Continuous Flow Water Monitor

The water is continuously injected at a tangent into a stationary flanged cylinder to cause rotation of that stream of water within a circular space of confinement. The injected stream of water becomes a rotating cylindrical wall of water of increasing volume and decreasing inside diameter, until the inside diameter of the water outlet (D) in the lower flange is reached and equilibrium is established. The inside diameter of the upper flange where the beta-gamma counter is inserted is smaller than the water outlet so as to prevent undesirable spill-over. For a water cell of dimensions as in Figure 2, a water flow of four liters per minute (water inlet pressure 20 psi) is required to maintain a wall of water of 11 mm radial thickness. Regulation of the water flow is not critical, and the counter wall is not touched by the water wall regardless of how slowly or rapidly the water flow is established.

Water Cell

The design includes several details (see Figure 2) to prevent turbulence in the water wall. The problem is one of continuously injecting water into a rotating cylindrical wall of water without disturbing the water-air boundary. For a given volume flow of water, a water jet having a circular cross-section would result in a minimum energy loss due to the friction of fluid with the jet wall. However, the circular jet introduces considerable turbulence in the water wall, and a slit jet (A) made by partial flattening of the water inlet tube provided a considerable improvement. The remaining amount of turbulence was largely eliminated by having a uniformly rising cam face (B) formed on one end of the cylinder (C), subtend the slit jet height over one revolution of the cylinder, thereby effectively introducing the jet stream not at a point

NOTE: DIMENSIONS ARE IN INCHES.

Fig.2 - Section View Of Water Cell

but throughout one revolution of the rotating water wall.

The choice of material for the water cell depends upon several factors, including the pH value, the particular isotopes involved, and the level of activity in the water. The removal of radioisotopes by plating out onto the water cell wall is increased if the water cell is metallic or if the water pH value is approximately 7, but is of no practical significance for the case of monitoring drinking water where the level of activity is usually less than about 1×10^{-8} $\mu\text{c/cc}$. A test to detect plate-out over an interval of five months has been made of the ORNL drinking water flowing into sample cells of different materials (brass and polyethylene) with negative results for each material. The sample cells in the dry condition were periodically measured for activity with a 20 kev beta sensitive Geiger counter (see Calibration Unit) in order that low energy beta or alpha radiation could be detected. Had radiation been detected in the dried sample cells, there still would not have been a significant increase in the background counting rate for the case of the cell containing the 11 mm thick water wall unless gamma radiation were present or beta particles of energy greater than about 2.1 mev. In practice, the 11 mm water wall is effective shielding for beta emitters of a maximum energy somewhat higher than 2.1 mev since the majority of betas emitted occur at about one-third of the maximum energy. Where the water flow to be monitored contains a gamma emitter of concentration greater than about 1×10^{-8} $\mu\text{c/cc}$, the water cell should be made of polyethylene or unplasticized polystyrene. The rise with time of the background counting rate due to plating out would depend upon the solution pH, and the amount of gamma emitting isotopes present and their

plating out characteristics. The addition of several drops of HCl per liter of water, to reduce the pH value to less than 2, would be particularly helpful in reducing plate-out. P^{32} is particularly troublesome in this respect, and it is difficult to remove that contamination from the cell wall.

Radiation Detector

The choice of a radiation detector (B) is influenced by the energy spectrum of the unstable isotopes present in the monitored liquid. If the activity includes mixed fission products or a significant percentage of beta emitters of about 0.7 mev maximum or higher, the radiation detector suggested is a thin wall GM counter such as the Radiation Counter Laboratory GM counter tube Mark 1, Model 11. Such a counter having a wall of about 30 mg/cm^2 will detect incident beta particles of energy greater than about 0.16 mev, and in practice is useful where the maximum energy of the beta particle is 0.5 mev or higher. Figure 3 is a graph of the sensitive region of a Model 11 GM counter tube, when the tube is scanned by a collimated beam of betas from $\text{Sr}^{90} + \text{Y}^{90}$. The background counting rate of the shielded counter is about 35 c/m.

Where detection of unstable isotopes emitting beta particles of less than about 0.7 mev maximum energy, or alpha particles is required, an alpha-beta counter (ORNL Dwg. No. D-9638) sensitive to incident betas of about 20 kev energy or higher is used. This is a cylindrical Geiger counter (Figure 4) with a 0.0002 in. thick wall (less than 0.7 mg/cm^2) of rubber hydrochloride, continuously flushed with 10% methane-90% argon counting gas at a flow rate of about 10 cc/min. The gas flow and pressure required to eliminate air from the counter will depend upon the size

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Fig.3-Geiger Counter Region Sensitivity (to Sr^{90} plus Y^{90})

FIG. 4 ALPHA-BETA COUNTER

and number of pin holes found in such thin rubber hydrochloride. Since the inner surface of the shell for electrical conductivity is coated with a transparent film of gold 50 microgram/cm², the counter is photosensitive and must be shielded from light. The shell is rugged and may be collapsed without injury. The Geiger region plateau and a region sensitivity graph are shown in Figure 5. The broken line graphs are the Geiger region slope and region sensitivity graphs for a non-uniform collector wire. Replacement of the wire resulted in the improved characteristics seen in the solid line graphs. Several replacements of the collector wire may be required to obtain a good Geiger region plateau. The background counting rate of the counter when shielded with three inches of lead and the water wall is approximately 10 c/m, and the useful beta window area is about 3.5 in.².

Operation of the beta counter requires a flow of counting gas at about 2 mm Hg differential pressure. The gas flow is metered (see Figure 6) with an oil manometer (A) measuring the pressure drop across an impedance (B) formed by a ten inch length of 0.031 in. inside diameter stainless steel tube. Using a gas bubbler may result in spurious counts due to pulsing of the counter shell. A pressure limiting device is formed by the inverted tube (C) which will permit gas to escape if the pressure rises above 5 mm Hg. Mechanical vacuum pump oil is used to avoid undesirable vapors. Fabrication of the gas control and some other parts must include careful internal cleaning followed by baking in a vacuum oven to remove water which seriously affects the characteristics of the counting gas. In practice, a counting gas flow of about 10 cc/min is adequate and this may be reduced if the counter shell is sufficiently gas-tight. When

Fig.5- 20-Kev Beta Counter

Fig.6 - Gas Flow Rate Meter

first applying pressure to the system, the connections between the gas flow controls, the gas supply tank, and the alpha-beta counter should be made cautiously after preliminary adjustments of the tank and regulator valves, to avoid blowing oil out or injuring the counter shell.

Calibration Unit

This instrument (Figure 7) is used to measure the response of a radiation detector to solutions of known activities and various maximum energies, with a geometry closely approximating that of the continuous flow water cell and detector assembly. A picture of the completed instrument is shown in Figure 7a. The response in c/m is plotted as a function of the radiation energy and activity, giving a graph of the sensitivity and energy dependence of the system.

The configuration of the continuous flow water cell and radiation detector is closely duplicated by an 80 cc liquid sample in a flanged sample cell of 131 cc total capacity (A), placed in a hollow rotor (B) fixed to an 1800 rpm motor shaft. Centrifugal force forms a vertical water wall of about one cm radial thickness, leaving an air space into which the alpha-beta counter (C) is lowered. Where solutions of alpha radiation or low energy beta particles are to be measured, a small volume cell is used as seen in Figure 8.

The beta energy dependence and sensitivity graph, curve A, shown in Figure 9, is for the alpha-beta counter described by ORNL Dwg. No. D-9638. The net counting rate is seen to be a linear function of the maximum beta energy between about 0.9 mev and 2.3 mev. Curve B describes the response of the Radiation Counter Laboratory GM tube Model 11, under substantially the same conditions. A temporary setup was used to accommodate the increased length of the commercial tube and is not described

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Fig. 7-Calibration Unit

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FIG. 7A ALPHA-BETA COUNTER, IN RAISED POSITION, READY FOR SAMPLE HANDLING. OBJECTS IN FOREGROUND ARE THE CONTINUOUS FLOW WATER CELL (LEFT) AND CALIBRATION UNIT SOLUTION CELL (RIGHT).

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BETA SOLUTION CELL

ALPHA SOLUTION CELL

MATERIAL
POLYETHYLENE

NOTE:
DIMENSIONS ARE IN INCHES

Fig.8— Calibration Unit Cells

Fig.9-Sensitivity and Energy Dependence
(calibration unit)

here since that tube would normally be used only in the continuous flow unit.

In practice, the calibration instrument is also useful for measuring radioactive liquid samples, of from 15 cc to 80 cc volume, where it is desirable to assess quickly the approximate activity of fluids. This is so because no time is lost in the evaporation to dryness of fluid samples as required for normal counting techniques. Where the identity of the unstable isotope is known, measurements of activity may be made between the approximate limits of 1×10^{-6} $\mu\text{c/cc}$ and 1×10^{-2} $\mu\text{c/cc}$, depending somewhat upon the energy of the particles. If the Calibration Unit is also calibrated with an absorber (for instance, a lucite cylinder of 0.015 in. wall thickness or 50 mg/cm^2 , sliding over the counter shell) a graph of N_1/N_2 plotted against maximum beta energy will permit assessing the approximate maximum energy of an unknown emitter (Figure 10).

EXPERIMENTAL RESULTS

The continuous flow water monitor was operated for several months, attached to a supply of drinking water. The possible contamination of that water supply would likely be by both high and low energy beta or gamma emitters; so the radiation detector used was a 30 mg/cm^2 wall Geiger counter (Radiation Counter Laboratory Model 11). The Geiger tube was connected to a commercial scaler (scale of 8) which fed into a Streeter-Amet Mechanical Recorder Model SCI-4. The recorder printed, at 15-minute intervals, the number of impulses received from the scaler during that time, and multiplying the recorder value by 8 gives the Geiger tube count with a recording error of not greater than 7 counts in 15 minutes. With a background counting rate of 35 c/m, the Geiger tube count

Fig.10 - Calibration Unit, Absorber Curves. A, 0.001 in. rubber hydrochloride absorber ; B, 0.015 in. lucite absorber ; N_1 = net c/m with absorber, N_2 = net c/m no absorber .

for 15 minutes would be 525; the recorder value would be 65; and the lost count or recording error would be $5/525$ or approximately 1%. The maximum integrating and recording error of the system is about 1.3%. The expected variations in the 15-minute background count are within $\pm 12\%$ of the average at the 0.99 confidence level; so it is feasible to calibrate the sensitivity of the monitor in terms of the overall response at the mechanical recorder. Threshold detection of activity is then a net count rate equal to 15% of the averaged background, and for a 1.5 mev beta emitter (see Figure 9) such as Sr^{89} would occur at a concentration of $3 \times 10^{-6} \mu\text{c/cc}$.

Where increased sensitivity is required for the detection of alpha particles or low energy beta radiation, the alpha-beta-gamma counter (ORNL Dwg. No. D-9638) is suggested. In general, the application of that counter is logically in the laboratory or equivalent surroundings because it is a continuous counting gas flow type. The hydrogenous gas (10% methane, 90% argon) results in a sensitivity to fast neutrons from a polonium-beryllium source such that a field of $12 \text{ n}_f/\text{cm}^2/\text{sec}$ gives a net count rate of 40 c/m. A field of $1 \text{ n}_f/\text{cm}^2/\text{sec}$ is detectable and would result in an assumed beta concentration. A radium source (0.18 mg in 1/2 mm platinum wall) at 50 cm distance gave a net counting rate of 32 c/m. The radium source at 50 cm distance, using the RCL Model 11 GM tube, gave a net counting rate of 30 c/m. The values cited are for the detectors in the 3 in. thick lead wall shield in normal operation.

The minimum amount of solution activity detectable is determined by the variations in the background counting rate. Radiation sources influencing the background count include: radiochemical

processing and reactor gaseous discharge, portable or transportable sources used in laboratory work, and the transporting of waste products. The monitor should be located at a considerable distance from an uncontrolled thoroughfare, and up-wind from radioactive gas discharges. As the wind direction does vary, large distances are desirable where a reactor site is involved.

Plating Out

The loss from a radioactive solution to the cell wall was measured with the Calibration Unit for $\text{Sr}^{90} + \text{Y}^{90}$ at several pH values and for two materials forming the cell wall. Plating out was minimized when the cell was machined from polyethylene and became insignificant when the solution pH was lowered to approximately one or two by the addition of HCl or HNO_3 .

Potassium⁴⁰ Measurements

Exposure of the human being to the principal radiation sources (cosmic rays, U-Th-Ac isotope series, Ra, K^{40}) occurring in nature, is assessed at 0.6 mrem/day.⁽⁴⁾ K^{40} is of particular interest because, as a fixed percentage component⁽⁵⁾ of natural potassium, it is present in the body fluids such as blood and urine and is available for test measurements by the liquid monitoring apparatus. However, an instrument capable of measuring directly the K^{40} concentration in body fluids must operate at a level comparable or lower than the background radiation exposure level of the human being.

The concentration of potassium in blood or urine is approximately 2 mg/gm.^(6,7) A water solution containing 2 mg of K per cc was measured in the Calibration Unit, giving a net counting rate of 5.4 ± 0.7 c/m.

APPENDIX

Construction of Alpha-Beta Counter

Letter size sheets of 0.0002 in. thick rubber hydrochloride are given a deposit of gold ($50 \text{ micrograms/cm}^2$) by evaporation on one face; resistivity of the coating is about five ohms per square and the characteristic green color of gold film is wrapped around a polished form (gold surface inside) and cemented along the lap joint with Tygobond No. 30 to form the thin shell (A) of the counter. A hot sheet formed end cap is cemented to the shell in a similar manner and the assembly air dried for three hours, then raised to 70° for ten minutes (to remove wrinkles), lowered to room temperature, and then blown (to minimize scratching of the gold film) off the form. The shell is assembled to the fitting (B) to be concentric, tied down with thread, and cemented. Stainless steel wire 0.001 in. diameter⁽⁸⁾ (C), selected for straightness and freedom from fibers is held in the connection pin (D) and the end weight (E) by means of the tapered pins. The connection pin, end weight, and tapered pins must be well polished, and the collector wire and tapered pin must terminate inside of the end weight. During assembly of the counter, the stainless steel collector wire is "dusted off" by lightly playing the discharge from a Tesla coil over it. The collector wire is grounded, while the Tesla coil is discharged to it, to avoid sparkover across insulator surfaces. If the counter shell is nominally gas-tight (this does not occur frequently), a pin hole about 0.001 in. diameter is made in the end cap (F) to permit flow of the counting gas.

APPARATUS DRAWING NUMBERS

(On file in the Engineering and Maintenance Division, ORNL)

Continuous Flow Water Monitor Assembly;	D-9029
Fig (lead shielding)	D-9030
Counter Gas Control Panel	D-9031
Water Cell (polyethylene)	D-9032
Water Cell (brass)	D-9034
GM Counter (commercial)	C-9650
20 Kev Beta Counter (ORNL)	D-9638
 Liquid Samples Beta Counter Assembly;	 D-9047
Fig (lead shielding)	D-9048
Counter Support	D-9049
Solution Cell and Motor Drive	D-9050
Motor Control Panel	C-9203
Counter Gas Control Panel	D-9031
20 Kev Beta Counter (ORNL)	D-9638

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